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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Thermal Treatment of Articles Composed of an Aluminum Base Alloy

We, ALUMINUM COMPANY OF AMERICA, a corporation organised under the laws of the State of Pennsylvania, United States of America, of Alcoa Building, Pittsburgh, Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the thermal treatment of articles of aluminum base alloys containing magnesium. More particularly, it is directed to a treatment for aluminum-magnesium alloys to prevent subsequent atmospheric and high-temperature oxidation and corrosion, and to facilitate degassing of finished and semi-finished articles of such alloys.

Thermal treatments, such as preheating the hot-working, annealing, solution heat-treatment and aging, are generally employed during the fabrication of products from aluminum and aluminium base alloys. These treatments are usually carried out in air atmosphere furnaces. In an air atmosphere which has not been dried and at temperatures above 800° F. but below the melting point of the alloy, aluminum-magnesium alloy articles tend to blister and stain, resulting in an objectionable appearance and sometimes a considerable decrease in mechanical strength. This effect is referred to as high-temperature oxidation.

Some finished and semi-finished articles of aluminum-magnesium alloys have been found to contain appreciable quantities of hydrogen, which may give rise to objectionable discontinuities in the metal structure. These discontinuities are generally quite easily detectable by ultrasonic testing techniques. It has been proposed to heat such articles in air for prolonged periods of time to diffuse hydrogen

from the metal into the surrounding atmosphere. However, it has been found that the presence of small quantities of moisture in the surrounding atmosphere results in the failure of such degassing. This failure to degas may be explained by reaction between the metal surface and moisture forming metal oxides and a high hydrogen partial pressure at the metal surface which prevents loss of hydrogen from the metal into the surrounding atmosphere. In some cases, the hydrogen partial pressure may be sufficiently high to cause additional gassing of the metal.

To minimize high temperature oxidation, various methods have been employed to protect these alloys at temperatures above 800° F. Specification 494,274 discloses the addition of fluorine-containing substances in vapor form to a non-dried air atmosphere to provide a protective environment for such alloys. The vapor is conveniently generated from solid compounds which decompose at the elevated temperatures, i.e., above 800° F. The metal is usually charged to a furnace already heated to the desired high temperature and containing the protective atmosphere or supplied with the vapor-generating fluoride compounds if the protective atmosphere has not been previously provided, and it is quickly heated to the desired temperature.

United States patent 2,379,467 discloses a protective treatment for aluminum alloy forgings in which an aqueous solution of sodium fluoroborate is applied to the forging which is then dried prior to being heated to the solution heat treating temperature. The coated forging is quickly heated to the required temperature in accordance with conventional practice.

Although these processes have been helpful in many cases, they have not eliminated altogether the undesirable blistering and staining which often occurs on articles of

aluminum-magnesium alloys, nor have they proven effective in reducing the gas content of the metal.

5 It has been found that the blistering and staining of such alloys is apparently related to the oxidation of magnesium atoms at the external surface of the article and that the particles of these oxides or other oxy-compounds act as focal points or channels for  
10 further oxidation on and within the surface. This oxidation is apparently caused by the attack of water upon the surface, oxidizing the aluminum and magnesium and releasing hydrogen.

15 It has further been determined that above a certain critical temperature, there is a rapid increase in the rate of magnesium atom diffusion to the surface and concomitant oxidation of the magnesium. Whereas in  
20 previous methods oxidation has been inhibited by providing a protective atmosphere above that critical temperature, it has now been discovered that, for complete protection of aluminum-magnesium alloys, they must be  
25 subjected to preliminary vapor treatment below that temperature, more particularly, at a point below the onset of any significant amount of magnesium oxidation.

30 It is an object of this invention to provide a preliminary thermal treatment for aluminum-magnesium alloy articles which prevents or inhibits high-temperature oxidation.

35 Another object is to provide a treatment for aluminum-magnesium alloy articles which will promote subsequent degassing of such articles in an atmosphere containing moisture.

40 It is also an object to provide a treatment for such articles which develops a surface condition that permits storage in industrial atmospheres for prolonged periods of time without corrosion.

45 A further object is to provide surface protection on aluminum-magnesium alloy articles which is stable at elevated temperatures in the presence of oxygen and moisture.

50 It has been discovered that the foregoing objects can be achieved by subjecting finished or semi-finished articles of the aluminum base alloys containing magnesium to a preliminary thermal treatment wherein they are exposed to an air or inert gas atmosphere containing at least 75 milligrams and preferably above  
55 270 milligrams of boron trifluoride per cubic foot of furnace atmosphere for a period of from one to forty-five minutes at a temperature of 400 to 750° F., but in all cases below the critical oxidation temperature of the alloy. Generally, it is preferred to heat the article to a temperature of above 400° F. but below  
60 575° F., the lowest critical oxidation temperature observed for an aluminum-magnesium

alloy. Although the atmosphere may consist predominantly of boron trifluoride vapors, yet for practical purposes it is preferred to use not more than 30 grams per cubic foot of  
65 furnace atmosphere so as to minimize problems of handling in commercial operations. The alloy articles which have been treated in this manner strongly resist oxidation at temperatures above 800° F. and up  
70 to the melting point of the lowest melting point constituent in the alloy which is often referred to as the temperature of incipient fusion. In addition, the treated articles may be degassed by subsequent heating for prolonged periods of times, even in the presence  
75 of moisture.

80 The critical oxidation temperature of an aluminum-magnesium alloy is defined as the lowest temperature at which the formation of magnesium oxide can be detected by electron diffraction techniques. This may be, in certain instances, a specific temperature or in other instances may constitute a narrow temperature  
85 band such as for example of approximately 20° F. Hence, this term is intended to cover both conditions. It is directly dependent upon the length of exposure in an untreated air atmosphere, the humidity of the furnace atmosphere, and the alloy composition,  
90 primarily the magnesium content. Although it can be determined readily for each set of conditions, it has been observed that these points fall within the range of 575 to 750° F., and generally 600 to 675° F.  
95

100 Generally, the electron diffraction determination involves heating samples in air to various temperatures and under various conditions of humidity and time. After such treatment, the samples are exposed to an electron beam in diffraction apparatus wherein the beam is reflected from the surface of the samples and a pattern is made upon a film as in X-ray diffraction studies. By comparing  
105 the pattern with that of a known sample, the nature of the substances on the surface of the metal sample can be determined. This diffraction pattern provides a qualitative indication of the presence of magnesium oxide.

110 The term "aluminum-magnesium alloy" as herein employed refers to an aluminum base alloy containing on a weight basis 0.1 to 15 per cent magnesium, with or without the presence of other elements, such as from 0.1 to 12 per cent copper or 0.25 to 14 per cent  
115 silicon or 0.1 to 20 per cent zinc or 0.1 to 3 per cent manganese, or combinations of two or more of these elements. Any of the foregoing alloys may also contain one or more of the following elements, often referred to as "hardeners," in the following percentages:  
120

	0.05	to 0.5	per cent chromium
	0.01	to 0.5	per cent titanium
	0.25	to 2.5	per cent nickel
5	0.01	to 0.5	per cent boron
	0.002	to 2	per cent beryllium
	0.1	to 0.5	per cent molybdenum
	0.1	to 0.5	per cent zirconium
	0.1	to 0.5	per cent tantalum
10	0.1	to 0.5	per cent columbium
	0.1	to 0.5	per cent cobalt

However, the total amount of the latter elements should not exceed 3 per cent.

The term "aluminum base alloy" as used herein refers to those compositions which contain at least 50 per cent by weight of aluminum.

The boron trifluoride-containing atmosphere may be provided by two methods. Solid inorganic carrier compounds may be deposited in a conventional air atmosphere furnace which, when heated to a temperature below the critical oxidation temperature of the alloy, will decompose or volatilize and yield boron trifluoride. As an alternative, the boron trifluoride vapor from a suitable source may be injected into the furnace atmosphere which has been heated to the desired temperature.

The inorganic carrier compounds should not yield the boron trifluoride below 400° F. since the protective film, if any, developed below this temperature is considerably less effective, possibly due to a different crystalline structure in the protective surface. The term "carrier" as used herein refers to those compounds which contain boron trifluoride in chemically combined or sorbed form but yield the free fluoride when heated above 400° F. but below 750° F. Examples of compounds found suitable as carriers are ammonium fluoroborate and calcium fluoroborate.

The above-described methods and compounds may be used singly or in combination.

The alloy articles are preferably exposed to the boron trifluoride-containing atmosphere for a period from one to forty-five minutes within the prescribed temperature range. Treatments of less than one minute do not permit development of a satisfactory protective film while treatments in excess of forty-five minutes do not provide any additional protection or offer any further benefit. Generally periods of five to fifteen minutes are sufficient; however, longer times of treatment are desirable in the case of alloys particularly susceptible to oxidation or when the surface oxide film is of greater thickness than that normally developed by exposure to the atmosphere.

The present process may be practiced in an untreated air atmosphere, i.e., a normal air atmosphere as commonly employed in heat-treating furnaces. No drying of the air need be undertaken as moisture can be

tolerated; in fact, the invention has been successfully employed even in furnace atmospheres having dew points of 125° F. Also, observations indicate that more satisfactory results are obtained when the furnace atmosphere contains at least  $\frac{1}{4}$  grain of water per cubic foot of furnace volume, but it should not exceed 45 grains per cubic foot. In addition, the atmosphere may be contaminated with such substances as sulfur dioxide, hydrogen chloride, ammonia, carbon monoxide and carbon dioxide without adverse effect upon the action of the boron trifluoride.

Gases which are inert toward the aluminum-magnesium alloys may be employed in place of air such as nitrogen, argon, helium and fuel gas. The term "atmosphere" as used herein includes air, the inert gases, or combinations thereof.

Further, this invention is most effectively practiced when the alloy articles have not previously been subjected to temperatures above their critical oxidation temperature. The presence of magnesium oxide on the surface, which has been produced in preceding thermal treatments, is observed to reduce the effectiveness of boron trifluoride, presumably due to the formation of oxy-fluoride complexes, but there is still benefit to be derived from the treatment of the present invention, even when alloy articles have been so pretreated.

When the inorganic boron-trifluoride carrier compounds are utilized, it is desirable to spread out the substance in a thin layer within the furnace so that a large surface area is exposed to heat and decomposition of the entire quantity may take place more readily.

The temperature employed for the subsequent degassing treatment should be above 750° F. but below the temperature of incipient fusion of the alloy. As is well-known, the higher the temperature the greater the rate of diffusion and the shorter the time required. Generally, for aluminium-magnesium alloys a temperature of from 900 to 975° F. has been found satisfactory.

The period for the degassing treatment will depend primarily on the thickness of the article being treated (the shortest diffusing path). Generally at 940° F. the heating should be continued for several hours and depending upon the thickness of the article, sometimes it is necessary to continue the treatment for as long as 20 days or more for articles having a maximum cross-section of  $\frac{1}{2}$  inch, periods in excess of 16 hours have been found adequate. A thickness of 1 inch will generally require a period of at least 40 hours at the same temperature; 10 to 15 days may be required for cross-sections thicker than 3 to 4 inches. Generally, the time required for the treatment will be related to the rate of hydrogen diffusion in the alloy at a given temperature.

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Subsequent to the degassing treatment, the treated article should be subjected to a working step to effect some reduction in size, thus healing the voids left by the diffused hydrogen.

5 The percentage of reduction necessary will be determined by the nature of the article and its original gaseous content.

The term "degassing," as used herein, contemplates both the removal of gas from the metal and the prevention of regassing of the metal.

The efficacy of the present invention is illustrated by the following examples:

#### EXAMPLE 1.

15 A lot of cold rolled sheets 0.064 inch in thickness of an alloy nominally composed of aluminum, 1.0 per cent magnesium, 0.6 per cent silicon, 0.3 per cent copper, 0.25 per cent chromium, and 0.06 per cent titanium, was divided into two groups for test purposes.

20 The first group was pretreated by exposure for a period of 10 minutes at a temperature of 575° F., which is below critical oxidation temperature, to an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate spread out on the floor of the furnace (30 grams being used per cubic foot of furnace volume). Subsequent to the pretreatment this

25 group of specimens was subdivided into three lots, each of which was heated at 1070° F. for a different period of time, namely,  $\frac{1}{2}$ , 32 and 64 hours, in an air atmosphere having a dew point of 125° F. The specimens were

quenched in water at the conclusion of the heat treatment. The second group, which was not pretreated, also was divided into three lots and each one was heated at 1070° F. for the same periods of time as the specimens in the first group. These specimens were

35 quenched in the same manner as those of the first group. All of the specimens of the first group were found to be free from blisters and staining, and metallographic examination indicated negligible traces of high temperature oxidation even after 64 hours. Such a long time of treatment is a severe test since it was much longer than the period of one to four hours normally employed for this alloy.

40 Visual examination of the second group, the untreated specimens, indicated that those exposed for only  $\frac{1}{2}$  hour exhibited light blistering and a grey-gold stain, and that upon longer exposure the oxidation increased; after 32 hours there was heavy blistering and a grey stain, and at the end of 64 hours, there was very severe blistering and a grey-black stain. Metallographic examination of the untreated specimens indicated the high temperature oxidation effects were moderate after  $\frac{1}{2}$  hour but heavy after 32 hours and very severe after 64 hours.

Specimens from each sub-division of the two groups were artificially aged at 350° F. for six hours in accordance with commercial practice and their tensile properties determined. The results are indicated in Table I below:

TABLE I

#### Mechanical Properties of Treated and Untreated Alloy Specimens

	Period of Treatment (Hrs.)	Tensile Strength (psi.)	Yield Strength (psi.)	Per Cent Elongation
Treated	1/2	50,300	44,400	12.8
	32	48,000	43,200	11.8
	64	48,400	43,300	11.3
Untreated	1/2	47,900	42,700	12.2
	32	42,900	40,200	5
	64	*	*	*

\* Specimens not capable of being tested because of severe deterioration.

The difference in mechanical properties of the treated and untreated alloy specimens appearing in Table I is significant and provides further evidence of the protective value of the treatment in a boron trifluoride containing atmosphere. High temperature oxidation brought about a complete loss in the mechanical properties of the untreated alloy over the longest period of exposure. In the absence of such oxidation the mechanical properties remained essentially constant after an early decrease of small proportions which may be attributable to the extreme severity of the conditions to which they were exposed. The reduction in elongation of the untreated lots exposed 32 and 64 hours is especially significant because it is considered to reflect rather closely the progress of penetration of the alloy by the undesired oxide. For this alloy in the artificially aged condition 10% elongation is considered to be the minimum value for structural purposes and yet the treated specimens exposed for 64 hours exceeded that minimum value.

#### EXAMPLE 2.

An alloy of the composition indicated in Example 1 and in the form of tube blooms was treated at 550° F. for a period of 30 minutes in an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate (25 grams per cubic foot of furnace volume). Both the treated and untreated specimens were exposed to an industrial atmosphere containing chlorine vapors for periods ranging from one to six weeks. Upon inspection, all of the treated blooms were found to be free from corrosion regardless of length of exposure whereas the untreated blooms showed a depth of attack of 0.0004 inch at the end of one week, 0.0008 inch at two weeks, 0.0010 inch at four weeks, and 0.0028 inch at six weeks.

#### EXAMPLE 3.

L-shaped extrusions of an alloy composed of aluminum, 2.5 per cent magnesium, 5.75 per cent zinc, 1.6 per cent copper, 0.24 per cent chromium, 0.05 per cent titanium, and 0.004 per cent beryllium were heated in a vertical air atmosphere solution heat-treating furnace to a temperature 450° F., which is below the critical oxidation temperature, at which temperature three pounds of boron trifluoride gas was injected into the undried furnace atmosphere (.09 gram boron trifluoride per cubic foot of furnace atmosphere). The boron trifluoride containing atmosphere was recirculated for about 30 minutes while the temperature of the

furnace was being elevated, and exhausted at about 650° F. by replacement with air. The specimens were subsequently heated to a solution heat treating temperature of 880° F. for a period of one hour and quenched in water. Visual inspection indicated no blistering or staining and metallographic examination disclosed negligible high temperature oxidation. Untreated specimens of the same alloy subjected to the above thermal treatment showed moderate blistering and a grey-black stain.

#### EXAMPLE 4.

Foil samples 0.003 inch in thickness of an alloy composed of aluminum, 1.5 per cent magnesium, 4.5 per cent copper, and 0.65 per cent manganese were divided into two groups. The first group of specimens was pretreated by exposure for about 15 minutes at a temperature of about 550° F. to boron trifluoride vapors generated by the decomposition of calcium fluoroborate (8 grams boron trifluoride per cubic foot furnace volume). The second group received no pretreatment. Both groups were heated to 920° F. for one hour in an air atmosphere having a dew point of 125° F. Inspection of the first group of treated specimens revealed no blistering or staining but the second or untreated group exhibited moderate blistering and had a grey-gold stain.

#### EXAMPLE 5.

Cold rolled sheets 0.064 inch in thickness of an alloy of the composition indicated in Example 1 were divided into two groups of specimens. The first group was pretreated by exposure for 15 minutes at a temperature of 500° F. in an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate (10 grams boron trifluoride per cubic foot of furnace volume) while the second group was not pretreated. The two groups were divided into lots, placed in a furnace and each lot in one group was heated at a temperature of 960° F. for a particular period of time ranging from  $\frac{1}{2}$  to 64 hours in length, as shown in Table II. All the specimens were quenched in water from the elevated temperature.

The specimens were subjected to metallographic examination and the degree or amount of high temperature oxidation was assigned an arbitrary numerical value from 0 to 10, 0 representing no oxidation or only a negligible amount while 10 represented the most severe condition. The untreated specimens exposed 32 and 64 hours were not rated because of very severe deterioration beyond the arbitrary scale. The results of the metallographic examination are indicated in Table II below:

TABLE II  
Metallographic Examination of Treated and Untreated Alloy Specimens

	Time of Exposure (Hrs.)	Amount of Oxidation
Treated	3/4	0
	1	0
	1—1/2	0
	4	0
	16	0
	64	0
Untreated	3/4	2
	1	3
	1—1/2	4
	4	5
	16	10
	32	Severe Deterioration
	64	Severe Deterioration

The protection afforded by the pretreatment is in striking contrast to the attack suffered by the untreated specimens, especially after a long period of exposure since that provided an extremely severe test. 25

#### EXAMPLE 6.

A lot of forgings of an alloy composed of aluminum, 4.3 per cent zinc, 3.3 per cent magnesium, 0.60 per cent copper, 0.2 per cent manganese, 0.18 per cent chromium and 0.06 per cent titanium was divided into two groups. The forgings each weighed approximately 600 pounds and varied in thickness from  $\frac{1}{2}$  inch to  $3\frac{1}{2}$  inches. The first group containing 37 forgings was pretreated by exposure for approximately 15 minutes at a temperature of approximately 500° F. in an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate deposited within the furnace (1.31 grams of boron trifluoride per cubic 20

foot of furnace volume). After pretreatment, the forgings were degassed by exposure to a temperature of 940° F. for 72 hours in an undried air atmosphere. The second group of 38 forgings was neither pretreated nor degassed. 25

Both groups were then subjected to a second blocking step and a finishing forging step, after which they were solution heat-treated at 830° F., quenched in water and subsequently precipitation hardening at 240° F. After heat treatment the forgings were ultrasonically inspected, the reject standard being an indication equal to or larger than that obtained from a No. 5 Series "B" Alcoa Ultrasonic Standard Reference Block of the appropriate metal distance. This method of testing is described in a technical paper by Panian and Van Valkenburg published in A.S.T.M. Vol. 59, 1959. The ultrasonic report on the first or treated group was as follows: 30 35 40

45  
32 pieces — clear (free from ultrasonic indications)  
1 piece — 1 (#3); 3 (#3+)  
2 pieces — 1 (#3); 4 (#3+); 2 (multiple #3)  
1 piece — 1 (#5)  
1 piece — 1 (#5); 2 (#5+)

or a total of 16 ultrasonic indications and a rejection of only two forgings. All of the forgings in the second group revealed ultrasonic indications and the group contained a total of 403 indications ranging from #3 or multiple #8+; from this group 22 forgings or 58 per cent were rejected.

The difference in the two groups indicated by the ultrasonic evaluation is most significant. That 32 of the forgings were completely free from indications is clearly convincing of the efficacy of the present inventions in facilitating degassing of aluminum-magnesium alloy articles.

#### EXAMPLE 7.

A lot of forgings of an alloy composed of aluminum, 4.4 per cent copper, 0.8 per cent silicon, 0.8 per cent manganese and 0.4 per cent magnesium was divided into two groups of 87 pieces each. The forgings each weighed about 115 pounds and varied in thickness between about  $\frac{3}{4}$  inch and 3 inches. The first group was pretreated by exposure for fifteen minutes at a temperature of approximately 500° F. in an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate (1.41 grams of boron trifluoride per cubic foot of furnace volume), and was then subjected to degassing at a temperature of 940° F. for about 72 hours in an undried air atmosphere. Both the degassed forgings and the untreated group were subjected to a blocking step and a finishing forging step after which they were solution heat-treated at 940° F., quenched in water, and then precipitation hardened at 340° F. Ultrasonic inspection indicated that the first group was substantially free from indications and all pieces passed inspection standards; the second group was found to contain numerous indications and 65 pieces were rejected for a recovery rate of only 25 per cent.

#### EXAMPLE 8.

A lot of forgings of the alloy described in Example 7 and which weighed about 135 pounds each and varied in thickness between about  $\frac{1}{2}$  inch and  $\frac{3}{4}$  inch were divided into two groups. The first group was pretreated by exposure for about fifteen minutes at a temperature of about 500° F. in an undried air atmosphere containing boron trifluoride generated by the decomposition of ammonium fluoroborate (1.41 grams of boron trifluoride per cubic foot of furnace volume). The pretreated specimens were then subjected to degassing at a temperature of 940° F. for about 72 hours in an undried air atmosphere. Both the degassed forgings and the untreated group were subjected to a second blocking step and

a finishing forging step, after which they were solution heat-treated at 940° F., quenched in water and precipitation hardened at 340° F. Upon inspection, the first group was found to be substantially free from ultrasonic indications and all pieces passed inspection standards while the second or untreated group contained numerous indications and only 50 per cent of the forgings met acceptance standards.

#### WHAT WE CLAIM IS:—

1. A method of treating a finished or semi-finished article composed of an aluminum base alloy containing from 0.1 to 15 per cent magnesium, which comprises exposing said article in a furnace to an atmosphere containing at least 75 milligrams of boron trifluoride vapor per cubic foot for a period of one to forty-five minutes at a temperature between 400° F. and 750° F. but below the critical oxidation temperature of said alloy.

2. A method according to claim 1, in which the article is exposed to said boron trifluoride-containing atmosphere at a temperature between 400 and 575° F.

3. A method according to claim 1 or 2, in which the atmosphere comprises air.

4. A method according to claim 3, in which the air contains at least  $\frac{1}{2}$  but not more than 45 grains of water per cubic foot.

5. A method according to any one of claims 1 to 4, in which the boron trifluoride vapor is provided by introducing boron trifluoride vapor into said atmosphere.

6. A method according to any of claims 1 to 4, in which the boron trifluoride vapor is provided by exposing to said atmosphere a solid inorganic boron trifluoride-containing compound which yields boron trifluoride at a temperature between 400° F. and 750° F.

7. A method according to any of claims 1 to 6, in which said article is subsequently degassed by thermal treatment at a temperature above 750° F. but below the temperature of incipient fusion.

8. A method of treating a finished or semi-finished article composed of an aluminum base alloy containing from 0.1 to 15 per cent magnesium according to claim 1 substantially as hereinbefore described, with reference to the Examples.

9. A finished or semi-finished article composed of an aluminum base alloy containing from 0.1 to 15 per cent magnesium when treated by the method according to any of claims 1 to 8.

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